PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : A23K 1/16, 1/18

A1

(11) International Publication Number: **WO 95/21540**

(43) International Publication Date: 17 August 1995 (17.08.95)

(21) International Application Number: PCT/US95/01510

(22) International Filing Date: 14 February 1995 (14.02.95)

(30) Priority Data:
196,333 14 February 1994 (14.02.94) US

(71) Applicant *(for all designated States except US)*: THE QUAKER OATS COMPANY [US/US]; 321 N. Clark Street, Chicago, IL 60610 (US).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: LEE, Patrick, S. [US/US]; Apartment #614, 249 N. Brand Boulevard, Glendale, CA 91203 (US). HAWLEY, Lawrence, A. [US/US]; 2292 S. Westwood Lane, Palatine, IL 60067 (US). KUNAS, Kurt, T. [US/US]; 447 S. Main Street, Wauconda, IL 60084 (US).

(74) Agents: JOHNSON, Lars, S. et al.; The Quaker Oats Company, Suite 25-7, P.O. Box 049001, Chicago, IL 60604-9001 (US).

(81) Designated States: AM, AT, AU (Petty patent), BB, BG, BR, BY, CA, CH, CN, CZ, DE, DE (Utility model), DK, ES, FI, GB, GE, HU, JP, KE, KG, KP, KR, KZ, LK, LT, LU, LV, MD, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SI, SK, TJ, TT, UA, US, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG), ARIPO patent (KE, MW, SD, SZ, UG).

Published
With international search report.
Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: ANIMAL FOOD PALATABILITY IMPROVING COMPOSITION AND PROCESS

(57) Abstract

The present invention relates to a process for preparing a composition for improving the palatability of animal food, said process comprising: (a) preparing an initial mixture comprising a proteinaceous material and a protein-hydrolyzing enzyme, wherein said protein-hydrolyzing enzyme comprises from 0.01 % to 0.5 % by weight of said initial mixture; (b) hydrolyzing the protein contained in the proteinaceous material of the initial mixture, thereby forming a hydrolyzed mixture, wherein said hydrolysis is carried out under conditions sufficient to hydrolyze a sufficient amount of protein to improve the palatability of an animal food when the composition resulting from step (f) of this process is combined with said animal food; (c) inactivating the protein-hydrolyzing enzyme contained in the hydrolyzed mixture, thereby forming an inactivated mixture; (d) heating or maintaining the inactivated mixture in an initial heating stage at a temperature in the range of from 80 °C to 100 °C for a period of time in the range of from 10 to 300 minutes; (e) following the initial heating stage, adjusting the pH of the inactivated mixture to within the range of from 2.8 to 3.8 while maintaining the temperature of the inactivated mixture in the range of from 80 °C to 100 °C, thereby forming an acidic mixture; and (f) heating or maintaining the acidic mixture in a final heating stage at a temperature and for a period of time sufficient to provide a composition which improves the palatability of animal food when combined therewith, thereby producing a palatability improving composition. The present invention still further relates to an animal food product having improved palatability, said product comprising animal food and an amount of said palatability improving composition sufficient to improve the palatability of said animal food. The present invention still further relates to a method of improving the palatability of animal food, said method comprising combining animal food with amounts of said palatability improving composition to improve the palatability of said animal food.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	**GB**	United Kingdom	**MR**	Mauritania
AU	Australia	**GE**	Georgia	**MW**	Malawi
BB	Barbados	**GN**	Guinea	**NE**	Niger
BE	Belgium	**GR**	Greece	**NL**	Netherlands
BF	Burkina Faso	**HU**	Hungary	**NO**	Norway
BG	Bulgaria	**IE**	Ireland	**NZ**	New Zealand
BJ	Benin	**IT**	Italy	**PL**	Poland
BR	Brazil	**JP**	Japan	**PT**	Portugal
BY	Belarus	**KE**	Kenya	**RO**	Romania
CA	Canada	**KG**	Kyrgyzstan	**RU**	Russian Federation
CF	Central African Republic	**KP**	Democratic People's Republic of Korea	**SD**	Sudan
CG	Congo			**SE**	Sweden
CH	Switzerland	**KR**	Republic of Korea	**SI**	Slovenia
CI	Côte d'Ivoire	**KZ**	Kazakhstan	**SK**	Slovakia
CM	Cameroon	**LI**	Liechtenstein	**SN**	Senegal
CN	China	**LK**	Sri Lanka	**TD**	Chad
CS	Czechoslovakia	**LU**	Luxembourg	**TG**	Togo
CZ	Czech Republic	**LV**	Latvia	**TJ**	Tajikistan
DE	Germany	**MC**	Monaco	**TT**	Trinidad and Tobago
DK	Denmark	**MD**	Republic of Moldova	**UA**	Ukraine
ES	Spain	**MG**	Madagascar	**US**	United States of America
FI	Finland	**ML**	Mali	**UZ**	Uzbekistan
FR	France	**MN**	Mongolia	**VN**	Viet Nam
GA	Gabon				

ANIMAL FOOD PALATABILITY IMPROVING COMPOSITION AND PROCESS

FIELD OF INVENTION

The present invention relates to a composition which, when applied to animal food, improves the flavor and the aroma, or palatability, of the animal food. The present invention also relates to the process for preparing said composition. The present invention further relates to an animal food product having an improved flavor and aroma, or palatability, due to its combination with said palatability improving composition.

BACKGROUND OF INVENTION

There is a continuing effort to develop processing and formulations which improve the palatability of animal food while maintaining its nutritional value. The improvement of palatability in animal food is important from at least two standpoints. First, it assures that the animal will consume a sufficient quantity of the food to maintain a healthy existence. Second, it enables the use of large amounts of basic food ingredients and by-products from the human food industry. This helps reduce the cost of human food production by providing a market for the by-products of this industry, while at the same time minimizing the demand of the animal feed industry for the choicer and more select raw materials which are suitable for human consumption. The present invention provides for such animal food having improved palatability.

BACKGROUND ART

There are numerous processes known in the art for improving and enhancing the palatability of animal food.

U.S. Patent 3,857,968, issued December 31, 1974, to Haas et al., discloses a process for improving the palatability of an animal food. The disclosed process comprises conditioning an aqueous meat slurry, comprising fat and protein, by emulsifying the fat in the meat slurry with the water present in the slurry, and treating the meat slurry, containing the emulsified fat and the protein, with an enzyme mixture comprises lipase and protease in

amounts effective to cause an enzymatic reaction resulting in the production of a palatability improving composition. Haas et al. also disclose incorporating this palatability improving composition into an animal food in an amount which is effective to increase the palatability of the animal food.

U.S. Patent 3,968,255, issued July 6, 1976, to Haas et al, discloses a process for improving the palatability of an animal food. The disclosed process comprises conditioning an aqueous meat slurry, comprising fat and protein, by emulsifying the fat in the meat slurry with the water present in the slurry, and treating the meat slurry, containing the emulsified fat and the protein, with lipase and protease in amounts, under conditions of pH and temperature, and for a time, effective to cause an enzymatic reaction resulting in the production of a palatability improving composition. Haas et al. also disclose incorporating this palatability improving composition into an animal food in an amount which is effective to increase the palatability of the animal food.

U.S. Patent 4,089,978, issued May 16, 1978, to Lugay et al., discloses a method for increasing the palatability of a nutritionally balanced dry dog food. The disclosed method comprises: (a) reacting an aqueous mixture of a reducing sugar, animal blood, fat, yeast or yeast extract with lipase and protease, at a temperature, pH, and for a time effective to cause lipolytic and proteolytic reaction; (b) heating the resulting aqueous reaction mixture to a temperature and for a time effective to inactivate the enzymes and to also further develop the palatability of the mixture, to produce a palatability enhancer and (c) applying an external coating of the palatability enhancer onto a dog food in an amount, between 1% and 5% based on the total weight of the dog food, which is effective to increase the palatability of the dog food.

U.S. Patent, 4,294,857, issued October 13, 1981, to Fuller, discloses a dog food composition having greater palatability to dogs than the same composition without the palatability enhancer. The dog food composition comprises a nutritionally balanced mixture of proteinaceous and farinaceous ingredients, and an added palatability enhancing 0.0001 to 0.01% by weight amount of linalool.

U.S. Patent 4,296,132, issued October 20, 1981, to Lazarus et al., discloses an improved nutritionally balanced dog food comprising fat, protein, carbohydrates, vitamins and minerals wherein the improvement consists of magnesium oleate in admixture with at least a portion of the fat, the magnesium oleate being present in an amount effective to improve the palatability of the dog food for dogs.

U.S. Patent 4,391,829, issued July 5, 1983, to Spradlin et al., discloses a process for preparing a nutritionally balanced dog food having improved palatability. The dog food comprises fat, protein, carbohydrates, vitamins and minerals and contains on a weight basis from 20% to 80% farinaceous material and from 20% to 80% proteinaceous ingredients, including proteinaceous meaty material at from 25% to 55% by weight of the proteinaceous ingredients. The disclosed process comprises the steps of (a) preparing a farinaceous slurry comprising from 30% to 70% water and from 5% to 25% of the total weight of said farinaceous ingredients; (b) forming a farinaceous reaction product by treating the farinaceous material in said farinaceous slurry with added alpha-amylase and protease enzymes in an amount and under conditions effective to convert at least a portion of the farinaceous material to a mixture of oligo saccharides ranging in size from 1 to 10 monomer units, and at lest a portion of the proteinaceous material to a mixture of peptides ranging in size from 2 to 300 monomer units, and effective to reduce the viscosity of the slurry by 50%; (c) preparing a proteinaceous slurry comprising water and a major portion of the proteinaceous meaty material; (d) forming a proteinaceous reaction product by treating the proteinaceous material in the proteinaceous slurry with protease enzyme in an amount and under conditions effective to convert at least a portion of the proteinaceous material to peptides ranging in size from 2 to 300 monomer units and effective to reduce the viscosity of the slurry by 50%; and (e) incorporating the farinaceous and proteinaceous reaction products into a dog food in an amount effective to provide a significant increase in the palatability of the dog food.

U.S. Patent 4,393,085, issued July 12, 1983, to Spradlin et al., discloses a process for preparing a nutritionally-balanced dog food having improved palatability. The dog food comprises fat, protein, carbohydrates, vitamins and minerals and contains on a weight basis

from 20% to 80% farinaceous ingredients and from 20% to 80% proteinaceous ingredients, including proteinaceous meaty material at from 25% to 55% by weight of the proteinaceous ingredients, said process comprising the steps of: (a) preparing a slurry comprising water, from 5% to 25% of the total weight of said farinaceous ingredients, and a major portion of the proteinaceous meaty material; (b) forming a reaction product by treating said slurry with added alpha-amylase and added protease enzymes in an amount and under conditions effective to convert at least a portion of the farinaceous material to a mixture of oligo saccharides ranging in size from 1 to 10 monomer units, and at least a portion of the proteinaceous material to a mixture of peptides ranging in size from 2 to 300 monomer units, and effective to reduce the viscosity of the slurry by 50%; and thereafter, (c) incorporating the reaction product of the slurry into a dog food in an amount effective to provide a significant increase in the palatability of the dog food to dogs.

U.S. Patent 4,713,250, issued December 15, 1987, to Tonyes et al., discloses a process for preparing a dog food palatability enhancer. The disclosed process comprises: (a) preparing a first stage reaction product by dispersing a substrate comprising a proteinaceous or farinaceous material in water and reacting the substrate with an enzyme or enzyme mixture comprising amylase when the substrate comprises farinaceous material and protease when the substrate comprises proteinaceous material, in amounts, under conditions of pH and temperature, and for a time effective to partially hydrolyze the substrate; (b) preparing an emulsion comprising fat and said first stage reaction product; and then (c) reacting said emulsion with lipase and protease in amounts, under conditions of pH and temperature, and for a time, effective to cause an enzyme reaction resulting in the production of a palatability enhancer.

U.S. Patent 4,804,549, issued February 14, 1989 to Howley et al., discloses a method of enhancing the palatability of an nutritionally balanced dog food with high levels of fats including high free fatty acids and which is resistant to package staining. The disclosed method compromises blending a high free fatty acids, and encapsulating binder comprising a protein or a carbohydrate, and water to from an emulsion. The emulsion is dried to produce a

palatability enhancer in flowable, particulate form. Pieces of dog food having exterior surfaces are then provided, and the palatability enhancer, which is in flowable particulate form, is mixed with the pieces of dog food.

U.S. Patent 5,186,964, issued February 16, 1993, to Gierhart et al., discloses a method of increasing the palatability of an extruded dry cat food composition. The disclosed process comprises topically applying to the cat food a palatability-improving composition consisting essentially of from 0.1 to 99%, by weight, of sodium acid pyrophosphate, in sufficient quantity to deposit from 0.5 to 2.0% of said sodium acid pyrophosphate, by weight of the food composition.

While there is extensive teaching in the art of methods to improve the palatability of animal food, there is nothing in the art which teaches the animal food palatability improving composition of the present invention, nor the process for its preparation.

It is therefore an object of the present invention to provide a process for preparing a composition which improves the palatability of animal food.

It is another object of the present invention to provide a process for producing said animal food palatability improving composition. It is still a further object of the present invention to provide an animal food having improved flavor and aroma, or palatability.

These objects are accomplished by the invention described herein. Unless otherwise specified, the percentages provided herein are weight percentages.

SUMMARY OF THE INVENTION

The present invention relates to a process for preparing a composition for improving the palatability of animal food, said process comprising:

(a) preparing an initial mixture comprising a proteinaceous material and a protein-hydrolyzing enzyme, wherein said protein-hydrolyzing enzyme comprises from 0.01% to 0.5% by weight of said initial mixture;

(b) hydrolyzing the protein contained in the proteinaceous material of the initial mixture, thereby forming a hydrolyzed mixture, wherein said hydrolysis is carried out under conditions sufficient to hydrolyze a sufficient amount of

protein to improve the palatability of an animal food when the composition resulting from step (f) of this process is combined with said animal food;

(c) inactivating the protein-hydrolyzing enzyme contained in the hydrolyzed mixture, thereby forming an inactivated-mixture;

(d) heating or maintaining the inactivated mixture in an initial heating stage at a temperature in the range of from 80°C to 100°C for a period of time in the range of from 10 to 300 minutes;

(e) following the initial heating stage, adjusting the pH of the inactivated mixture to within the range of from 2.8 to 3.8 while maintaining the temperature of the inactivated mixture in the range of from 80°C to 100°C, thereby forming an acidic mixture; and

(f) heating or maintaining the acidic mixture in a final heating stage at a temperature and for a period of time sufficient to provide a composition which improves the palatability of animal food when combined therewith, thereby producing a palatability improving composition.

The present invention further relates to the palatability improving composition produced by said process.

The present invention still further relates to an animal food product having improved palatability, said product comprising animal food and an amount of said palatability improving composition sufficient to improve the palatability of said animal food.

The present invention still further relates to a method of improving the palatability of animal food, said method comprising combining animal food with amounts of said palatability improving composition to improve the palatability of said animal food.

DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a process for preparing a composition which improves the palatability of animal foods, and preferably improves the palatability of pet foods such as dog food. The present invention also provides a palatability improving composition prepared by the process of the present invention.

In the process of the present invention an initial mixture comprising a proteinaceous material and a protein-hydrolyzing enzyme is prepared. The proteinaceous material is one that provides a positive palatability response when hydrolyzed, and is preferably capable of being emulsified and is available in quantity at a reasonable cost. Any proteinaceous material known to those skilled in the art as providing such a positive palatability response may be used in the present innovation. Examples of such proteinaceous materials useful in the present invention include, but are not limited to, meat, soy products, whey, chicken skins, cheese, gluten, meat-by-products, animal offal, poultry-by-products, brewer's yeast, and mixtures thereof, with meat being preferred.

Any meat source known to those skilled in the art may be used in the present invention. Examples of meat sources useful herein include, but are not limited to, beef liver, deboned beef meat, pork liver, chicken livers and parts, chicken viscera, beef, pork, chicken, turkey, fish, and mixtures thereof.

Especially preferred are proteinaceous materials which additionally provide all or part of the protein-hydrolyzing enzyme also contained in the initial mixture, such as chicken viscera. Preferred is a combination of from 30% to 70%, more preferably from 40% to 60% by weight chicken viscera, and from 30% to 70%, more preferably from 40% to 60% by weight beef livers, wherein said weight percentages are based on the weight of the initial mixture.

The initial mixture of the present invention also comprises a protein-hydrolyzing enzyme. This protein-hydrolyzing enzyme may be provided to the initial mixture either by the addition of a purified sample of a protein-hydrolyzing enzyme, or, as already stated herein, may be included as an indigenous component of the proteinaceous material added to the initial mixture. One skilled in the art will appreciate that the proteinaceous material and protein-hydrolyzing enzyme are combined in a manner to allow for sufficient hydrolysis of the proteinaceous material.

Any protein-hydrolyzing enzyme known to those skilled in the art may be used in the present invention. Examples of useful protein-hydrolyzing enzymes include, but are not

limited to, protease enzymes. It is currently contemplated that any protease enzyme derived from plants, animals, or microorganisms, can be employed in the present invention, as long as it is capable of providing the requisite improvement in palatability. Examples of protease enzymes useful herein include, but are not limited to, plant proteases such as bromolein and papain, proteases derived from *B.cuptilis*, and animal derived enzymes such as trypsin and pepsin. A preferred protease is Alkalase 0.6 L enzyme, which is manufactured by the Enzyme Process Division of Bioindustrial Group, Novo Nordisk A/S, located in Denmark. As far as is known, Novo Nordisk has a sales office located in Danbury, CT. According to Novo Nordisk's sales literature, Alkalase 0.6 L is produced from a selected strain of *Bacillus licheniformis*. The main enzyme component, *Subtilisin A* (= *Subtilisin Carlsberg*), is an endoproteinase, which is extensively described in the literature.

While the degree of enzymatic action of the protein-hydrolyzing enzyme is dependent upon a number of factors, the protein-hydrolyzing enzymes will typically be employed in an amount and for a period of time effective to provide generation of desired polypeptides to achieve palatability improving properties in the final product of the process of the present invention. The protein-hydrolyzing enzyme typically comprises from 0% to 1%, preferably from 0.01% to 0.75%, more preferably from 0.1% to 0.5% by weight of the initial mixture.

The various protein-hydrolyzing enzymes come in a wide variety of commercial forms, including dry, purified enzymes; enzymes supported on various soluble and insoluble carriers; and enzyme solutions of various strengths.

The initial mixture is typically prepared by combining the proteinaceous material with the protein-hydrolyzing enzyme, and this could be done by any manner known to those skilled in the art. For ease of handling, especially in view of the effect of those materials in body tissues, it is preferred to add the protein-hydrolyzing enzyme to the initial mixture in the form of a liquid solution. In addition, water may be added to the initial mixture to allow improved mixing of the proteinaceous material and enzyme.

After the initial mixture is prepared, it is reacted to prepare a hydrolyzed mixture. The initial mixture is reacted under conditions sufficient to hydrolyze a sufficient amount of

protein available in the initial mixture to impart palatability-improving properties to the composition product of the process of the present invention. Preferably at least 85%, more preferably at least 90%, still more preferably at least 95% by weight of the protein available in the initial mixture is hydrolyzed.

The protein hydrolysis is typically carried out at a temperature in the range of from 40°C to 75°C, preferably from 45°C to 75°C, still more preferably from 55°C to 65°C, for a period of time in the range of from 5 to 240, preferably from 15 to 120, still more preferably from 30 to 120 minutes. When adding heat to carry out the protein hydrolysis, care should be taken to avoid inactivating the protein-hydrolyzing enzyme. Also, as will be appreciated by one skilled in the art, if steam is used to supply heat for the hydrolysis reaction, the steam may condense, which in turn may contribute water to the initial mixture.

After the protein present in the initial mixture is sufficiently hydrolyzed, a hydrolyzed mixture is formed. The protein-hydrolyzing enzyme contained in the hydrolyzed mixture is then substantially inactivated, thereby forming an inactivated mixture. The protein-hydrolyzing enzyme is preferably inactivated by heating the hydrolyzed mixture to a temperature sufficient to inactivate the particular protein-hydrolyzing enzyme. For the preferred protein-hydrolyzing enzyme Alkalase 0.6 L, this is accomplished by heating the hydrolyzed mixture to a temperature of at least 80°C, preferably at least 85°C, still more preferably at least 90°C.

Other methods of inactivating the protein-hydrolyzing enzyme may be used, although care must be taken to avoid inactivating any protein-hydrolyzing enzymes through adjustment of the pH. The point in which the pH is adjusted in the present invention is critical, and any early adjustment of the pH will have an adverse effect on the product of a process of the present invention, i.e., will reduce its palatability improving effectiveness.

The inactivated mixture is then heated in an initial heating stage. In this initial heating stage the inactivated mixture is heated to a temperature in the range of from 80°C to 100°C, preferably from 85°C to 95°C, more preferably from 87°C to 92°C, for a period of time in a range of from 10 to 300 minutes, preferably from 15 to 120 minutes, still more

preferably from 30 to 60 minutes. This initial heating stage may also involve maintaining the temperature of the initial mixture at an already elevated level, for example by insulating the reaction vessel.

Following the initial heating stage, the pH of the inactivated mixture is adjusted to within the range of from 2.5 to 3.8, preferably from 2.7 to 3.5, still more preferably from 2.9 to 3.2, thereby forming an acidic mixture. The pH may be adjusted by the addition of any edible acidifying agents known to those skilled in the art. Examples of acidifying agents useful in the present invention include, but are not limited to, phosphoric acid and hydrochloric acid, with phosphoric acid being preferred.

Of course, the amount of acidifying agent added to the inactivated mixture will be known to those skilled in the art as that amount necessary to achieve the desired pH.

The adjustment of the pH of the inactivated mixture is carried out in conjunction with heating. The temperature of the inactivated mixture during the pH adjustment is maintained in the range of from 70°C to 100°C, more preferably from 80°C to 90°C.

The acidic mixture is then heated in a final heating stage at a temperature and for a period of time sufficient to provide a composition which improves the palatability of animal food when combined therewith. In this final heating stage the acidic mixture is preferably heated at a temperature in the range of from 75°C to 99°C, more preferably from 80°C to 95°C, still more preferably from 85°C to 90°C, for a period of time in the range of from 1 to 24 hours, more preferably from 5 to 15 hours, still more preferably from 8 to 12 hours. As with the initial heating stage, this final heating stage may also involve maintaining the temperature of the acidic mixture at an already elevated level, for example by insulating the reaction vessel.

The final palatability improving composition prepared by this process has been shown to improve the palatability of animal feed when combined with said animal feed. While not intending to be bound by theory, it is surprising and unexpected that the particular heating, pH adjustment, and further heating of the hydrolyzed proteinaceous material of the present invention, in the sequence provided in the present invention, can provide a palatability-

improving composition. It has been found that this palatability-improving composition has palatability-improving properties superior to compositions containing hydrolyzed proteinaceous material prepared by methods which do not include the particular pH and heating sequence of present invention.

In a more preferred embodiment, in the process of the present invention the initial heating stage, the acidic mixture forming stage, and the final heating stage are carried out in the presence of air.

By the term "in the presence of", it is meant that air is brought into contact with the respective mixtures of each of these stages. The air may be brought into contact with the respective mixtures by any means known to those skilled in the art, for example by bubbling the air through the respective mixtures. Note should be taken, however, that is not preferred to have the reaction exposed to the open atmosphere. This diminishes the performance of the palatability-improving composition prepared by the process of the present invention. It is believed that this diminishment in performance is caused by the volatilization of flavor-improving components produced by the process of the present invention. Preferred is when the reaction is carried in out in an enclosed vessel with excess air present.

While not intending to be bound by theory, it is believed that the oxygen present in the air acts to oxidize off-odor agents contained in the respective mixtures. As will be appreciated by one skilled in the art, the necessary amount of air is that amount sufficient to oxidize the off-odor reagents present in the respective mixtures.

In a still more preferred embodiment of the present invention, the process for preparing the palatability improving composition comprises:

(a) preparing an initial mixture comprising from 40% to 60% by weight chicken viscera, from 40% to 60% by weight beef livers, and from 0.1% to 0.5% by weight protease, wherein said weight percentage are expressed as a percentage of the initial mixture;

(b) hydrolyzing the protein contained in the chicken viscera and beef livers by reacting the protease with said protein in the presence of air and at a

temperature in the range of from 55°C to 65°C for a period of time in the range of from 30 to 120 minutes, thereby forming a hydrolyzed mixture;

(c) inactivating the protease in the hydrolyzed mixture by heating the hydrolyzed mixture to a temperature of at least 90°C, thereby forming an inactivated mixture;

(d) heating or maintaining the inactivated mixture in an initial heating stage to a temperature in the range of from 87°C to 92°C for a period of time in the range of from 30 to 60 minutes;

(e) following the initial heating stage, adjusting the pH of the inactivated mixture to within the range of from 2.9 to 3.2 while maintaining the temperature of the inactivated mixture in the range of from 80°C to 90°C, thereby forming an acidic mixture; and

(f) heating or maintaining the acidic mixture in a final heating stage at a temperature in the range of from 85°C to 90°C for a period of time in the range of from 8 to 12 hours, thereby forming a palatability-improving composition.

The present invention further relates to a product for improving the palatability of animal food, wherein said product is prepared by the process comprising:

(a) preparing an initial mixture comprising a proteinaceous material and a protein-hydrolyzing enzyme, wherein said protein-hydrolyzing enzyme comprises from 0.01% to 0.5% by weight of said initial mixture;

(b) hydrolyzing the protein contained in the proteinaceous material of the initial mixture, thereby forming a hydrolyzed mixture, wherein said hydrolysis is carried out under conditions sufficient to hydrolyze a sufficient amount of protein to improve the palatability of an animal food when the composition resulting from step (f) of this process is combined with said animal food;

(c) inactivating the protein-hydrolyzing enzyme contained in the hydrolyzed mixture, thereby forming an inactivated-mixture;

(d) heating or maintaining the inactivated mixture in an initial heating stage at a temperature in the range of from 80°C to 100°C for a period of time in the range of from 10 to 300 minutes;

(e) following the initial heating stage, adjusting the pH of the inactivated mixture to within the range of from 2.8 to 3.8 while maintaining the temperature of the inactivated mixture in the range of from 80°C to 100°C, thereby forming an acidic mixture; and

(f) heating or maintaining the acidic mixture in a final heating stage at a temperature and for a period of time sufficient to provide a composition which improves the palatability of animal food when combined therewith, thereby producing a palatability improving composition.

The present invention also provides for an animal food composition having improved palatability, wherein said animal food composition comprises animal food and a palatability-improving composition. The palatability improving composition is prepared by the process comprising:

(a) preparing an initial mixture comprising a proteinaceous material and a protein-hydrolyzing enzyme, wherein said protein-hydrolyzing enzyme comprises from 0.01% to 0.5% by weight of said initial mixture;

(b) hydrolyzing the protein contained in the proteinaceous material of the initial mixture, thereby forming a hydrolyzed mixture, wherein said hydrolysis is carried out under conditions sufficient to hydrolyze a sufficient amount of protein to improve the palatability of an animal food when the composition resulting from step (f) of this process is combined with said animal food;

(c) inactivating the protein-hydrolyzing enzyme contained in the hydrolyzed mixture, thereby forming an inactivated-mixture;

(d) heating or maintaining the inactivated mixture in an initial heating stage at a temperature in the range of from 80°C to 100°C for a period of time in the range of from 10 to 300 minutes;

(e) following the initial heating stage, adjusting the pH of the inactivated mixture to within the range of from 2.8 to 3.8 while maintaining the temperature of the inactivated mixture in the range of from 80°C to 100°C, thereby forming an acidic mixture; and

(f) heating or maintaining the acidic mixture in a final heating stage at a temperature and for a period of time sufficient to provide a composition which improves the palatability of animal food when combined therewith, thereby producing a palatability improving composition.

A preferred animal feed composition according to the present invention comprises animal food and a palatability-improving composition, wherein the palatability-improving composition is prepared by the process comprising:

(a) preparing an initial mixture comprising from 40% to 60% by weight chicken viscera, from 40% to 60% by weight beef livers, and from 0.1% to 0.5% by weight protease, wherein said weight percentage are expressed as a percentage of the initial mixture;

(b) hydrolyzing the protein contained in the chicken viscera and beef livers by reacting the protease with said protein in the presence of air and at a temperature in the range of from 55°C to 65°C for a period of time in the range of from 30 to 120 minutes, thereby forming a hydrolyzed mixture;

(c) inactivating the protease in the hydrolyzed mixture by heating the hydrolyzed mixture to a temperature of at least 90°C, thereby forming an inactivated mixture;

(d) heating or maintaining the inactivated mixture in an initial heating stage to a temperature in the range of from 87°C to 92°C for a period of time in the range of from 30 to 60 minutes;

(e) following the initial heating stage, adjusting the pH of the inactivated mixture to within the range of from 2.9 to 3.2 while maintaining the temperature of the

inactivated mixture in the range of from 80°C to 90°C, thereby forming an acidic mixture; and

(f) heating or maintaining the acidic mixture in a final heating stage at a temperature in the range of from 85°C to 90°C for a period of time in the range of from 8 to 12 hours, thereby forming a palatability-improving composition.

The animal feed composition comprises animal feed, preferably from 85% to 98%, more preferably from 90% to 97%, still more preferably from 95% to 97% by weight animal feed, and a sufficient amount of palatability-improving composition to improve the palatability of the animal feed, preferably from 1% to 15%, more preferably from 2% to 10%, still more preferably from 2.5% to 5% by weight palatability-improving composition.

The present invention further provides a process for improving the palatability of an animal food composition. The process comprises:

(a) initially preparing a palatability-enhancing composition by the process comprising:

(1) preparing an initial mixture comprising a proteinaceous material and a protein-hydrolyzing enzyme, wherein said protein-hydrolyzing enzyme comprises from 0.01% to 0.5% by weight of said initial mixture;

(2) hydrolyzing the protein contained in the proteinaceous material of the initial mixture, thereby forming a hydrolyzed mixture, wherein said hydrolysis is carried out under conditions sufficient to hydrolyze a sufficient amount of protein to improve the palatability of an animal food when the composition resulting from step (a)(6) of this process is combined with said animal food;

(3) inactivating the protein-hydrolyzing enzyme contained in the hydrolyzed mixture, thereby forming an inactivated-mixture;

(4) heating or maintaining the inactivated mixture in an initial heating stage at a temperature in the range of from 80°C to 100°C for a period of time in the range of from 10 to 300 minutes;

(5) following the initial heating stage, adjusting the pH of the inactivated mixture to within the range of from 2.8 to 3.8 while maintaining the temperature of the inactivated mixture in the range of from 80°C to 100°C, thereby forming an acidic mixture;

(6) heating or maintaining the acidic mixture in a final heating stage at a temperature and for a period of time sufficient to provide a composition which improves the palatability of animal food when combined therewith, thereby producing a palatability improving composition; and

(b) incorporating the palatability-improving composition into the animal food in an amount effective to increase the palatability.

The final animal food/palatability-improving composition mixture is as already described herein.

The palatability-improving composition can be incorporated into the animal food in any suitable matter. Thus, where the complete animal food or portion thereof is conditioned, the palatability-improving composition is incorporated by producing it *in situ*. Or, the palatability-improving composition can be prepared separately from the animal food and then mixed with or applied to the animal food.

Application of the palatability-improving composition to dry animal food by spraying is preferred for dry animal food because it allows uniform surface application without breaking the emulsion. This makes it possible to obtain improved palatability with significantly smaller amounts of the palatability-improving composition. One suitable device for spraying the emulsion to the animal food is a spray nozzle of the kind commonly employed in spray painting.

The palatability-improving composition can be in an emulsified state for incorporation into the animal food. In fact, when initially prepared by the process described herein, it is contained in an emulsified state.

The palatability-improving composition prepared in this matter is generally applied in any effective amount to improve the palatability of the animal food. It has been found in practice that amounts as low as 0.1% by weight, based on the total weight on the animal food, has provided sufficient improvement in palatability for dogs. Generally, amounts of greater than 5% of weight based on the total weight of the animal food are not employed unless the animal food so treated is later diluted with another material such as untreated animal food, meats scraps, water, or the like.

Another method of incorporating the palatability-improving composition of the present invention is to mix it directly with other ingredients in the preparations of the animal food. For example, the palatability-improving composition is admixed with other ingredients of animal food such as fat, carbohydrates, protein, vitamins and minerals, in any effective amount to provide an animal food of improved palatability. The palatability-improving composition can generally comprise from 1 to 35%, preferably from 5 to 20%, of the animal food. As a guideline to an upper limit, it is noted that high concentrations seem to adversely affect the texture of the animal food, and should be avoided where the impairment of the texture outweighs the improvement in palatability.

While the palatability-improving composition prepared according to the present invention can improve the palatability of animal foods generally, they are especially suitable for use with nutritionally balanced comprising protein, fat, carbohydrates, vitamins and minerals.

The present invention is further illustrated, but not limited by, the following examples.

EXAMPLES

Example 1

A mixture of meat is prepared by grinding to a 0.32 cm. size the following material: (1) 34.02 kg. chicken viscera, (2) 6.80 kg. mechanically deboned beef, and (3) 6.80 kg. of

beef liver. This mixture is conveyed to a jacketed vessel of sufficient volume to hold the mixture to an 80% capacity The material is heated to 60°C by circulating a hot water bath in the jacket of the vessel. The material is agitated during this heating period.

When the mixture temperature reaches 60°C, 0.23 kg. of Alkalase 0.6L enzyme is added to the mixture. While maintaining a temperature of 60°C, the mixture is continuously agitated by a rotating scraped surface agitator as well as by being pumped continuously through a recirculating shear pump. The agitation and recirculation assure that the mixture is well mixed.

After 60 minutes of heating and agitation, steam is circulated through the jacket of the vessel to heat the mixture to a temperature of 90°C. The mixture temperature is maintained at this level for 60 minutes. This inactivates the enzyme and preheats the mixture for the next processing step. At this point the mixture is in the form of what is commonly referred to as a "digest."

The mixture is next allowed to cool moderately while being pumped to another closed vessel. This holding vessel is sufficiently large to hold the mixture to a capacity of no more than 80% full. The mixture is allowed to cool to a temperature of 70°C. 1.81 kg. of Phosphoric acid is added to the mixture, which adjusts the mixture pH to 3.0 While the acid is added, the mixture is continuously agitated by a scrape-surface agitator. The total acid addition time is no more than 15 minutes.

After the acid is added, the mixture is heated to a temperature of 85°C. The 85°C temperature is maintained for a period of 10 hours by circulating hot water around the jacket of the holding vessel. The mixture is agitated during the 10 hour heating period.

The mixture is next sprayed onto pet food kibbles in a continuous-flowing coating reel. The digest is added along with choice white grease at a 1:1 ratio, and this mixture is added to dry kibbles at a total concentration of 5%. The final kibble formula has a digest concentration of 2.5%, a grease level of 2.5%, a moisture of 10% and a dry solids content of approximately 90%.

Example 2

A mixture of meat is prepared by grinding to a 0.32 cm. size the following material: (1) 38.56 kg. poultry-by-product, (2) 6.80 kg. of beef liver. This mixture is conveyed to a jacketed vessel of sufficient volume to hold the mixture to an 80% capacity The material is heated to 60°C by circulating a hot water bath in the jacket of the vessel. The material is agitated during this heating period.

When the mixture temperature reaches 60°C, 0.23 kg. of Alkalase 0.6L enzyme is added to the mixture. While maintaining a temperature of 60°C, the mixture is continuously agitated by a rotating scraped surface agitator as well as by being pumped continuously through a recirculating shear pump. The agitation and recirculation assure that the mixture is well mixed.

After 60 minutes of heating and agitation, steam is circulated through the jacket of the vessel to heat the mixture to a temperature of 90°C. The mixture temperature is maintained at this level for 30 minutes. This inactivates the enzyme and preheats the mixture for the next processing step. At this point the mixture is in the form of what is commonly referred to as a "digest."

The mixture is next allowed to cool moderately while being pumped to another closed vessel. This holding vessel is sufficiently large to hold the mixture to a capacity of no more than 80% full. The mixture is allowed to cool to a temperature of 70°C. 1.81 kg. of Phosphoric acid is added to the mixture, which adjusts the mixture pH to 3.0 While the acid is added, the mixture is continuously agitated by a scrape-surface agitator. The total acid addition time is no more than 15 minutes.

After the acid is added, the mixture is heated to a temperature of 85°C. The 85°C temperature is maintained for a period of 5 hours by circulating hot water around the jacket of the holding vessel. The mixture is agitated during the 5 hour heating period.

The mixture is next sprayed onto pet food kibbles in a continuous-flowing coating reel. The digest is added along with choice white grease at a 5:6 ratio, and this mixture is added to dry kibbles at a total concentration of 11%. The final kibble formula has a digest

concentration of 5%, a grease level of 6%, a moisture of 10% and a dry solids content of approximately 90%.

Example 3

A mixture of meat is prepared by grinding to a 0.32 cm. size the following material: (1) 34.02 kg. chicken viscera, (2) 11.34 kg. of beef liver. This mixture is conveyed to a jacketed vessel of sufficient volume to hold the mixture to an 80% capacity The material is heated to 60°C by circulating a hot water bath in the jacket of the vessel. The material is agitated during this heating period.

When the mixture temperature reaches 60°C, 0.05 kg. of Alkalase 0.6L enzyme is added to the mixture. While maintaining a temperature of 60°C, the mixture is continuously agitated by a rotating scraped surface agitator as well as by being pumped continuously through a recirculating shear pump. The agitation and recirculation assure that the mixture is well mixed.

After 30 minutes of heating and agitation, steam is circulated through the jacket of the vessel to heat the mixture to a temperature of 90°C. The mixture temperature is maintained at this level for 30 minutes. This inactivates the enzyme and preheats the mixture for the next processing step. At this point the mixture is in the form of what is commonly referred to as a "digest."

The mixture is next allowed to cool moderately while being pumped to another closed vessel. This holding vessel is sufficiently large to hold the mixture to a capacity of no more than 80% full. The mixture is allowed to cool to a temperature of 70°C. 1.81 kg. of Phosphoric acid is added to the mixture, which adjusts the mixture pH to 3.0 While the acid is added, the mixture is continuously agitated by a scrape-surface agitator. The total acid addition time is no more than 15 minutes.

After the acid is added, the mixture is heated to a temperature of 85°C. The 85°C temperature is maintained for a period of 7 hours by circulating hot water around the jacket of the holding vessel. The mixture is agitated during the 5 hour heating period.

The mixture is next sprayed onto pet food kibbles in a continuous-flowing coating reel. The digest is added along with choice white grease at a 1:1 ratio, and this mixture is added to dry kibbles at a total concentration of 5%. The final kibble formula has a digest concentration of 2.5%, a grease level of 2.5%, a moisture of 10% and a dry solids content of approximately 90%.

Example 4

A mixture of meat is prepared by grinding to a 0.32 cm. size 22.68 kg. chicken necks and backs and 22.68 kg. of mechanically deboned beef. This mixture is conveyed to a jacketed vessel of sufficient volume to hold the mixture to an 80% capacity The material is heated to 60°C by circulating a hot water bath in the jacket of the vessel. The material is agitated during this heating period.

When the mixture temperature reaches 60°C, 0.23 kg. of Alkalase 0.6L enzyme is added to the mixture. While maintaining a temperature of 60°C, the mixture is continuously agitated by a rotating scraped surface agitator, as well as by being pumped continuously through a recirculating shear pump. The agitation and recirculation assure that the mixture is well mixed.

After 60 minutes of heating and agitation, steam is circulated through the jacket of the vessel to heat the mixture to a temperature of 90°C. The mixture temperature is maintained at this level for 10 minutes. This inactivates the enzyme and preheats the mixture for the next processing step. At this point the mixture is in the form of what is commonly referred to as a "digest."

The mixture is next allowed to cool moderately while being pumped to another closed vessel. This holding vessel is sufficiently large to hold the mixture to a capacity of no more than 80% full. The mixture is allowed to cool to a temperature of 70°C. 0.23 kg. of Phosphoric acid is added to the mixture, which lowers the mixture pH slightly to approximately 6.0 While the acid is added, the mixture is continuously agitated by a scrape-surface agitator. The total acid addition time is no more than 15 minutes.

After the acid is added, the mixture is heated to a temperature of 85°C. The 85°C temperature is maintained for a period of 1/2 hour by circulating hot water around the jacket of the holding vessel. The mixture is agitated during the 1/2 hour heating period. This acidified digest is pumped continuously from the holding tank to a extrusion process as described below.

The acidified digest is added continuously to a preconditioner. A dry mixture consisting of 40% ground corn, 27% wheat, 27% soy, and 6% other vitamin and mineral materials, is also fed to the preconditioner, along with the acidified digest. The ratio of acidified digest to dry mixture being fed to the preconditioner is approximately 1:4 (acidified digest:dry mixture). As the dry mixture is blended with the acidified digest, water and steam are added to the contents of the preconditioner. This brings the total moisture of the mixture to 20 to 25% (including the moisture which exists as part of the dry mixture's natural content). The mean residence time of the resulting wet dough in the preconditioner is approximately 2 to 5 minutes.

The wet dough is next transferred to an Anderson extruder. The extruder has a 7.62 cm. diameter and a typical screw profile suitable for extruding pet food. Water and steam are added to the extrudate in the barrel of the extruder. The extruder screw rotates at 150 rpm and produces kibbles at a rate of 250 kg. per hour. The final extrudate has a moisture content of 25 to 35% (wet basis) and a temperature of no less than 120°C.

Kibbles are produced by extruding the wet dough through a die consisting of 10 openings, all of which have the same shape. As the dough exits the extruder through the die, it is pelleted continuously by a fly-knife cutter. Kibbles exiting the extruder are transferred directly to a dryer, where they are dried to a moisture of 8% to 10% wet basis.

The dry kibbles are next enrobed with a mixture of the digest and choice white grease. The digest and choice white grease mixture is sprayed onto the kibbles in a continuous-flowing coating reel. The ratio of digest to choice white grease is 1:1, and this mixture is added to dry kibbles at a total concentration of 5%. The final kibble formula has a digest

concentration of 2.5%, a grease level of 2.5%, a moisture content of 10% and a dry solids content of approximately 90%.

WHAT IS CLAIMED IS:

1. A process for preparing a composition for improving the palatability of animal food, said process comprising:

(a) preparing an initial mixture comprising a proteinaceous material and a protein-hydrolyzing enzyme, wherein said protein-hydrolyzing enzyme comprises from 0.01% to 0.5% by weight of said initial mixture;

(b) hydrolyzing the protein contained in the proteinaceous material of the initial mixture, thereby forming a hydrolyzed mixture, wherein said hydrolysis is carried out under conditions sufficient to hydrolyze a sufficient amount of protein to improve the palatability of an animal food when the composition resulting from step (f) of this process is combined with said animal food;

(c) inactivating the protein-hydrolyzing enzyme contained in the hydrolyzed mixture, thereby forming an inactivated-mixture;

(d) heating or maintaining the inactivated mixture in an initial heating stage at a temperature in the range of from 80°C to 100°C for a period of time in the range of from 10 to 300 minutes;

(e) following the initial heating stage, adjusting the pH of the inactivated mixture to within the range of from 2.8 to 3.8 while maintaining the temperature of the inactivated mixture in the range of from 80°C to 100°C, thereby forming an acidic mixture; and

(f) heating or maintaining the acidic mixture in a final heating stage at a temperature and for a period of time sufficient to provide a composition which improves the palatability of animal food when combined therewith, thereby producing a palatability improving composition.

2. A process according to claim 1 wherein the proteinaceous material is selected from the group consisting of meat, soy products, whey, chicken skins, cheese, gluten, meat by-products, animal offal, poultry by-products, brewer's yeast, and mixtures thereof; wherein the

protein-hydrolyzing enzyme is provided to the initial mixture by the addition of protease; wherein the protein-hydrolyzing enzyme comprises from 0.01% to 0.75% by weight of the initial mixture; wherein the initial mixture is reacted at a temperature in the range of from 45°C to 75°C for a period of time in the range of from 15 to 120 minutes; wherein the protein-hydrolyzing enzyme contained in the hydrolyzed mixture is inactivated by heating the hydrolyzed mixture to a temperature of at least 85°C; wherein the inactivated mixture is heated in the initial heating stage at a temperature in the range of from 85°C to 95°C for a period of time in the range of from 15 to 120 minutes; wherein the pH of the acidic mixture is within the range of from 2.7 to 3.5 while maintaining the temperature of the inactivated mixture in the range of from 80°C to 90°C; and wherein the acidic mixture is heated in the final heating stage at a temperature in the range of from 80°C to 95°C for a period of time in the range of from 5 to 15 hours.

3. A process for preparing a composition for improving the palatability of animal food, said process comprising:

(a) preparing an initial mixture comprising from 40% to 60% by weight chicken viscera, from 40% to 60% by weight beef livers, and from 0.1% to 0.5% by weight protease, wherein said weight percentage are expressed as a percentage of the initial mixture;

(b) hydrolyzing the protein contained in the chicken viscera and beef livers by reacting the protease with said protein in the presence of air and at a temperature in the range of from 55°C to 65°C for a period of time in the range of from 30 to 120 minutes, thereby forming a hydrolyzed mixture;

(c) inactivating the protease in the hydrolyzed mixture by heating the hydrolyzed mixture to a temperature of at least 90°C, thereby forming an inactivated mixture;

(d) heating or maintaining the inactivated mixture in an initial heating stage to a temperature in the range of from 87°C to 92°C for a period of time in the range of from 30 to 60 minutes;

(e) following the initial heating stage, adjusting the pH of the inactivated mixture to within the range of from 2.9 to 3.2 while maintaining the temperature of the inactivated mixture in the range of from 80°C to 90°C, thereby forming an acidic mixture; and

(f) heating or maintaining the acidic mixture in a final heating stage at a temperature in the range of from 85°C to 90°C for a period of time in the range of from 8 to 12 hours, thereby forming a palatability-improving composition.

4. A process for improving the palatability of an animal food which comprises:

(a) initially preparing a palatability-enhancing composition by the process comprising:

(1) preparing an initial mixture comprising a proteinaceous material and a protein-hydrolyzing enzyme, wherein said protein-hydrolyzing enzyme comprises from 0.01% to 0.5% by weight of said initial mixture;

(2) hydrolyzing the protein contained in the proteinaceous material of the initial mixture, thereby forming a hydrolyzed mixture, wherein said hydrolysis is carried out under conditions sufficient to hydrolyze a sufficient amount of protein to improve the palatability of an animal food when the composition resulting from step (a)(6) of this process is combined with said animal food;

(3) inactivating the protein-hydrolyzing enzyme contained in the hydrolyzed mixture, thereby forming an inactivated-mixture;

(4) heating or maintaining the inactivated mixture in an initial heating stage at a temperature in the range of from 80°C to 100°C for a period of time in the range of from 10 to 300 minutes;

(5) following the initial heating stage, adjusting the pH of the inactivated mixture to within the range of from 2.8 to 3.8 while maintaining the temperature of the inactivated mixture in the range of from 80°C to 100°C, thereby forming an acidic mixture;

(6) heating or maintaining the acidic mixture in a final heating stage at a temperature and for a period of time sufficient to provide a composition which improves the palatability of animal food when combined therewith, thereby producing a palatability improving composition; and

(b) incorporating the palatability-enhancing composition into the animal food in an amount effective to increase the palatability of the animal food.

5. A process according to claim 4 wherein the animal food is a dog food; wherein the palatability-enhancing composition is incorporated into the animal food by admixing it in-situ with other ingredients of animal food such as fat, carbohydrates, protein, vitamins and minerals, in any effective amount to provide an animal food of improved palatability; and wherein the palatability improving composition employed in the amount of from 5% to 20%, based on the weight of the animal food.

INTERNATIONAL SEARCH REPORT

Intern al Application No
PCT/US 95/01510

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A23K1/16 A23K1/18

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A23K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	GB,A,1 583 644 (SPILLERS LIMITED) 28 January 1981 see page 2, line 23 - line 47 see page 3, line 8 - line 26 see page 9, line 37 - line 38 see claim 1	1-5
Y	US,A,4 089 978 (JOAQUIN CASTRO LUGAY ET AL.) 16 May 1978 cited in the application see column 5, line 19 - line 37	1-5
A	US,A,4 731 248 (WILLIAM C. HOGAN ET AL.) 15 March 1988 see column 2, line 32 - column 3, line 5 see column 5, line 3 - line 15 see column 5, line 22 - column 6, line 36 see examples 6,8,10	1-5

-/--

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
7 June 1995	05. 07. 95

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax (+ 31-70) 340-3016	Dekeirel, M

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Interm al Application No
PCT/US 95/01510

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US,A,3 876 806 (WALTER L. HEMPENIUS ET AL.) 8 April 1975 see column 2, line 16 - line 48 see column 4, line 9 - line 43 see column 5, line 61 - column 6, line 16 see example I	1

1

Form PCT/ISA/210 (continuation of second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

information on patent family members

Interr al Application No
PCT/US 95/01510

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
GB-A-1583644	28-01-81	NONE		
US-A-4089978	16-05-78	BE-A-	852802	18-07-77
		CA-A-	1063857	09-10-79
		CH-A-	604557	15-09-78
		DE-A-	2708816	13-10-77
		FR-A-	2357187	03-02-78
		GB-A-	1537431	29-12-78
		JP-C-	1273252	11-07-85
		JP-A-	52117781	03-10-77
		JP-B-	59050300	07-12-84
		NL-A-	7702578	04-10-77
US-A-4731248	15-03-88	NONE		
US-A-3876806	08-04-75	CA-A-	974397	16-09-75
		FR-A-	2202652	10-05-74
		GB-A-	1363950	21-08-74